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TWENTY-FIRST PROGRESS REPORT

to

National Aeronautics and Space Administration

on

Cryogenic Research and Development

for

Period Ending March 31, 1966



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Cryogenics Division
Institute for Materials Research
National Bureau of Standards
Boulder, Colorado

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1. Physical Properties of Cryogenic Fluids

1.0 General Comments

Personnel contributing to the activities during this period were: R. D. Goodwin, W. J. Hall, H. M. Roder, L. A. Weber, and B. A. Younglove.

1.1 Parahydrogen

1.1.1 Dielectric Constant of the Solid

Continued difficulty in freezing a sample in the dielectric cell was experienced due to freezing in the filling capillary. Installation of a heater on the capillary was ineffective and, consequently, the capillary was relocated so as to enter the bomb near the bottom rather than at the top where the proximity of cooling due to the support tube was thought to be troublesome. Further tests of sample freezing are now in progress.

1.1.2 Index of Refraction

For the past seven months, an apparatus has been under development for the measurement of the refractive index of gases and liquids. The most important design criterion is that it shall be possible to examine the density and temperature dependence of the Lorenz-Lorentz function, $LL \equiv \frac{n^2 - 1}{n^2 + 2} \cdot \frac{1}{\rho}$, for parahydrogen. From previous measurements of the dielectric constant, it is anticipated that L-L for hydrogen will be constant to about 0.2% over a very large range of temperatures and densities. To examine the small remaining dependence, we plan to measure refractive index with a precision of 0.0002%. Our knowledge of density as derived from measurement of pressure and temperature and reference to the $\rho(P, T)$ data published by this Laboratory will have a precision of 0.02%.

To obtain refractive index measurements with the above precision, we have constructed a small Fabry-Perot interferometer which

can be placed in a high pressure bomb. The interferometer consists of two semi-transparent surfaces separated by a cylindrical tungsten spacer 0.5 cm in length and 1.5 cm outside diameter. Tungsten was chosen to minimize the change in length when the spacer is hydrostatically compressed. Two quartz optical flats coated with aluminum to provide the semi-transparent surfaces are wrung on the ends of the spacer which have been lapped flat and parallel to a few millionths of an inch. When monochromatic light of wave length, λ , is passed through the interferometer, a circular fringe pattern can be observed. If a gas is admitted to the interferometer, the fringe pattern will change with the density of the gas. The number of fringes, ΔN , passing a reference point as the sample is attenuated to a vacuum may then be counted. Refractive index can be computed from the fringe count using the expression

$$n - 1 = \frac{\lambda (\Delta N)}{2t}$$

where n = refractive index and t = spacer thickness.

Sufficient apparatus has been assembled to begin test measurements at room temperature. A cryostat for low temperature measurements is also near completion. A mercury 198 lamp is used to provide monochromatic light of well-known wave length. The integral fringe count is determined by a photomultiplier and recorder. The center of the fringe pattern is focused on a pinhole in front of the photomultiplier. The fractional fringe count is measured by photographing the fringe pattern with a spectrograph followed by measurements of the diameters of the images of three adjacent rings with a travelling microscope. Using these methods, ΔN can be determined with a precision of 0.02 fringe.

The refractive index of air at ambient conditions was measured as a simple first test. The result, $n = 1.0002200 \pm 0.0000004$, agreed favorably with a value, $n = 1.0002202$, obtained some years ago by

Barrell and Sears, NPL, from much more precise measurements. Tests are now in progress on nitrogen gas at room temperature and pressures to 150 atm. The results can be compared with published measurements of Michels et al.(1949). Some difficulty has been experienced with uncertainty in the fringe count caused by interaction of the pressure measuring device with the gas in the high pressure cell. Improved skill of the operator can apparently minimize this problem. Tests on argon and hydrogen gas at room temperature are also planned before completing assembly of the cryostat and electrical wiring of the high pressure bomb.

1.1.3 Thermal Conductivity

Development and testing of the experimental apparatus has been going on with NBS support. Several modifications have been made which can be understood by referring to the rather complete description of the apparatus in the First Quarterly Report. These have been as follows:

- a) The hot-plate thickness and the heights of the two guards have been reduced. This change reduces the thermal lag and the correction for the change of enthalpy of these members when slow drifts of temperature have not been completely eliminated.
- b) The cold plate was removed, and the inside bottom of the bomb was refinished so that it now serves as the cold plate. Difficulty had been experienced in maintaining good thermal contact between the original cold plate and the bomb.
- c) The hot plate and guard heaters were moved from the side surfaces to interior locations in the interest of obtaining more uniform heat distribution in those parts.
- d) The spacers were located under the auxiliary guard, and the guard-hot plate assembly was supported by three radial pins mounted in the wall of the auxiliary guard. As energy accounting

now shows, this has greatly improved the uniformity of heat dissipation by the guard and this presumably has reduced fringing at the edge of the hot plate and local temperature inhomogeneities in the guard that may have interfered with obtaining a thermal null balance between the guard and hot plate.

e) The temperature controls have been more fully automated.

f) A shielded lead has been installed that permits measuring the hot plate to cold plate capacitance, using the thermal guard member as an electrical guard. Knowing the dielectric constant of the medium, the cell constant can be calculated from the capacitance. This provides a monitoring of effective cell geometry which is superior to reliance on prior gaging of plate and spacers because it can detect changes due to slippage of the radial support pins and accounts for fringing.

g) A larger tube to the bomb has been installed. This permits more effective evacuation and hence a more accurate evaluation of radiation corrections.

h) The bomb cooling coils and cooling block have been removed in order to improve the rate of response of the bomb to its surface-mounted heaters.

The present phase of the system is best termed "shake down" experiments. In the last four months we have tried to determine the following:

a) Is the system operating correctly? Are all modes of heat flow in the system and its container understood?

b) What is the estimate of error?

c) What is the best mode for routine operation?

These experiments have been made with helium, air, and carbon dioxide at about atmospheric pressure where either published

data are believed to be reliable or values can be calculated rather reliably from kinetic theory.

RESULTS:

Preliminary values of thermal conductivity for He gas at pressures near one atmosphere and temperatures between 80 and 300°K have been established. The results are analyzed in terms of deviation from basic kinetic theory.

$$K = 2.5 \cdot \eta_{\text{meas}} \cdot C_v$$

where $\eta_{\text{meas}} = 5.023 \cdot T^{0.647}$ (equation 2.84 reference [1])

and $C_v = 3.116 \text{ joule/gram}^\circ\text{K}$ (equation 2.70 reference [1])

The results run from 2% low at 80°K to 1.5% high at 300°K.

A limited number of measurements made on CO₂ near 300°K and one atmosphere are 0.4% higher than those of Sengers [2].

EXPERIMENTS AND DETAILS:

a) The basic equation used is that due to Fourier

$$K = \frac{\dot{Q} \Delta x}{A \Delta T} \quad \text{which changes to } K = \frac{\dot{Q} f}{C \Delta T} \quad (\text{see 2 below})$$

where $\dot{Q} = \dot{Q}_{\text{meas}} - \dot{Q}_{\text{radiation}} \pm \dot{Q}_{\text{drift of hot plate}}$

b) The cell constant, $\Delta x / A$, is determined by a capacitance measurement. The main elements of the conductivity system form a guarded Kelvin capacitor for which the capacitance is

$$C = \frac{f A}{\Delta x} \quad \text{where } f \text{ is a numerical factor.}$$

For a careful room temperature assembly, the capacitance as measured agrees to 3 parts/20,000 with that calculated from the dimensions of the parts. The capacitance has a slight variation

with temperature. Proper accounting of the dimensional changes of hot plate (copper) and alignment pins (plastic) permits calculation of $C(T)$ which closely approximates the values observed. The plastic pins used to support the hot plate permit a slight displacement with respect to the guard due to differential thermal contractions. Experiments with a capacitor mockup show that the capacitance measurement adequately accounts for any fringing that may occur.

c) The radiation contribution to the heat flow was measured in vacuum near room temperature. The result of 0.205 mW/degree of temperature difference compares to 0.147 mW/degree of temperature difference calculated from basic equations assuming a value of 0.018 for the emissivity of copper. The change in emissivity is almost identical to that experienced by Sengers [2].

d) The temperature difference is measured with two platinum resistance thermometers. Measurements with zero heat input establish that one or both thermometers have changed somewhat from their calibration. To make these "zero" runs, three heaters are required to keep the high pressure vessel completely isothermal. Under normal operating conditions the beryllium-copper bomb experiences a slight gradient in temperature that depends on the hot plate power input. Since the "cold plate" resistance thermometer is separated from the cold plate surface by a substantial thickness of beryllium-copper, a correction for the temperature difference across this metal is calculated. Thus,

$$\Delta T = \Delta T_{\text{measured}} + \Delta T_{\text{calibration shift}} - \Delta T_{\text{Be-Cu}}$$

e) For the present experimental conditions K varies little with temperature. A valuable check on internal consistency is to observe the variation, if any, of K with power input. Considerable effort has been placed in this area and if all corrections are applied properly, K is found to be independent of power input. In the second type

of test the guard is operated with a known mismatch in temperature. For an offset of $\pm 0.004^{\circ}\text{K}$ the conductivity changes by $\pm 2\%$. Finally, because the interior guard is not supported by a solid member, the power dissipation can be used to establish a value of thermal conductivity from the guard geometry. It is extremely reassuring that values so obtained differ by less than 2% from the values measured for the hot plate.

f) Estimates of Error. Contributions arising from the value of \dot{Q} are less than 0.1%. The control of the guard is to $0.03\mu\text{V}$. For temperatures above 80°K and ΔT of 1 degree this is equivalent to 0.3%. Errors from the measurement of the cell factor are less than 0.1%. Errors in the value of ΔT used depend on the value of ΔT and on the conductivity of the fluid under test. As examples for:

He gas with $\Delta T = 0.2^{\circ}\text{K}$ the error is 0.85%

He gas with $\Delta T = 0.85^{\circ}\text{K}$ the error is 0.5%

He gas with $\Delta T = 1.7^{\circ}\text{K}$ the error is 0.5%

CO_2 gas with $\Delta T = 2.0^{\circ}\text{K}$ the error is 0.2%

The absolute error is a statistical combination, thus for He gas with ΔT of 1°K or larger, we get 0.6%; and for the measurements of CO_2 , we get 0.4%. The precision or reproducibility of experiments is governed mainly by the control and zero of the guard and seems to be around 0.2%.

FORTHCOMING TESTS OF THE APPARATUS:

Using data recording sheets and a computer program already developed, test experiments on He gas at pressures of 1, 3, and 10 atmospheres in the temperature range $20\text{-}300^{\circ}\text{K}$ are contemplated.

[1] Helium, editor, Keesom, Elsevier (1942).

[2] Sengers, J. V. Thesis, University of Amsterdam (1962).

1.1.4 Equation of State. The equation of state already mentioned (20th Progress Report) has been greatly improved as compared with results in NBS Report 9100. The equation has been constrained to both the accepted triple point and the accepted critical point. At the same time, the mean deviation from data has diminished. It has been necessary to introduce three characteristic constants and three coefficients, all concerned with the critical point, such that the total number of constants is 25. Whereas best values for some of these constants are obtained only by costly, iterative procedures, we believe the basic structure for this equation of state now has been found.

1.1.5 TAB Code. The corrections listed in the last report for codes in the 19th Progress Report contained errors. Following is a restatement of these corrections:

PTVISCCH beginning in card col. 7

PTVISC=(FP*FT*V(I)+F*FT*V(I+1)+FP*FF*V(J)+F*FF*V(J+1))*10.0

PTCOND29 beginning in card col. 7

DATA((C(I),I=175,188)=.20584, .20584, .20584, .20587, .20592, .20705,

PTCOND30 beginning in card col. 6

1.20926, .36287, .35682, .35199, .35150, .35489, .35610, .35634)

PTCONDBU beginning in card col. 6

8.56859, 2.1303, 1.2148, 1.0124, .88843, .70784, .62686, .60582, 2.8357,

PTDENS DM beginning in card col. 6

6,.000137,.09778,.1928,.2850,.000111,.08837,.1745,.2584,.000092,.0

As a preliminary to setting up the specific heat TAB Code, the specific heat for high temperatures is being recalculated using the spectroscopic data as reported in NBS Monograph 20 (May 1961).

Two new transport property codes are given in this report. The two new codes, PHVISC and PHCOND, both have pressure, P, ranges from 1 to 5000 psia and enthalpy, H, ranges from -130 to 20,000 BTU/lb. These codes are based on the correlations done by Los

Alamos as presented in their report LASL 2527 and revised in LASL 2719. For viscosity, Diller's experimental data* were used in the temperature range from 14° to 100°K instead of the correlation. The codes return values which are within 1% of the source data. Pressures which are out of the range of the codes are extrapolated from the last values in the range. Values of enthalpy below the solidus line will give the values at the solidus line. Values above 20,000 BTU/lb will give the value at 20,000.

The Fortran statement:

VISCOS=PHVISC(P,H)

would cause the viscosity in lb-hr/ft² at P and H to be stored in the location VISCOS. Similarly:

CONduc=PHCOND(P,H)

would return the thermal conductivity in BTU/hr-ft-°R to CONduc. The program listings are as follows:

*Dwain E. Diller, Measurements of the viscosity of Parahydrogen, J. Chem. Phys. 42, No. 6, 2089-2100 (March 1965).

```

FUNCTION PHCOND(PRES,ENTH)
COMMON/PHLAMB/C(802)
DIMENSION LOC(18),JP(18),DP(18),DH(18),BP(18),BH(18),MX(18),
IPL(19),HL(19),HG(19),CL(19),CG(19),HS(11)
DATA(LOC=1,64,78,123,147,175,205,245,295,335,395,430,480,500,608,
1660,715,779)
DATA(JP=7,7,9,6,7,6,8,10,8,5,7,5,5,9,4,5,8,6)
DATA(MX=5,5,7,4,5,4,6,8,6,3,5,3,3,7,2,3,6,4)
DATA(BP=0,0,-200.,500.,0,0,-50.,500.,-50.,4000.,1000.,3000.,1000.,
11000.,400.,0,50.,0)
DATA(BH=9600.,4500.,500.,425.,425.,225.,225.,125.,125.,15.,65.,-25
1.,20.,-90.,-115.,-130.,20.,65.)
DATA(DP=0,0,400.,500.,100.,1000.,100.,500.,100.,250.,500.,250.,500
1.,250.,200.,100.,50.,10.)
DATA(DH=1300.,5100.,1000.,200.,200.,50.,50.,25.,25.,10.,15.,10.,15
1.,10.,20.,15.,15.,20.)
DATA(PL=1.022,2.,4.,8.,14.,25.,43.,69.,99.,128.,151.,165.,176.,182
1.,185.,186.5,187.25,187.46875,187.506)
DATA(HL=-132.8,-129.13,-124.25,-117.79,-110.86,-101.3,-89.04,
1-74.22,-58.58,-43.43,-30.07,-20.56,-11.13,-4.27,1.17,5.54,10.83,
214.29,16.36)
DATA(HG=60.31,65.11,70.59,76.35,80.98,85.11,87.40,86.54,81.94,
174.15,64.83,56.86,47.34,39.56,33.46,28.34,22.31,18.66,16.55)
DATA(HS=-132.82,-113.35,-94.24,-75.47,-57.04,-38.91,-21.05,-3.4,
114.08,31.36,48.53)
DATA(CL=.07328,.07187,.07093,.06899,.06787,.06665,.06522,.06338,
1.06115,.05865,.05614,.05403,.05174,.04974,.04765,.04596,.04515,
2.04509,.04442)
DATA(CG=.006439,.007004,.007737,.008716,.009848,.011681,.01467,
1.01925,.02476,.03005,.03404,.03649,.03852,.04005,.04159,.04296,
2.04369,.04391,.04442)
DATA((C(I),I=430,499)=.1257,.145,0,0,0,.1116,.1304,.15,0,0,.1012,
1.11545,.1354,.1556,0,.09387,.1045,.1194,.1399,.161,.08875,.09667,
2.1079,.12345,.1447,.08516,.09111,.09957,.1114,.1276,.08265,.08717,
3.09358,.1026,.115,.08092,.08438,.08927,.09616,.1057,.07971,.08242,
4.08619,.09148,.09884,.07888,.08106,.08400,.08809,.09378,.06586,
5.07074,.07436,.07869,.08679,.06463,.06996,.07364,.07715,.08265,
6.06338,.06918,.07305,.07623,.08026,.06214,.06840,.07251,.07564,
7.07888)
DATA((C(I),I=500,607)=.08142,.09051,.1016,0,0,0,0,0,0,.07697,
1.08407,.09425,.109,0,0,0,0,0,.07426,.07911,.08685,.09818,.1094,0,
2.0,0,0,.07253,.07603,.08136,.08975,.1021,.1148,0,0,0,.07135,.07408
3,.07789,.08372,.09275,.1061,.1195,0,0,.07045,.07277,.07566,.07984,
4.08619,.09589,.11015,.1217,0,.06968,.07184,.0742,.07731,.08188,
5.08878,.09913,.11631,.13080,.06894,.07110,.07319,.07565,.07904,
6.08402,.09147,.10248,.11843,.06820,.07044,.07244,.07453,.07715,
7.08084,.08627,.09429,.10593,.06744,.06982,.07182,.07373,.07588,
8.07870,.08273,.08862,.09721,.06666,.06920,.07127,.07312,.07500,
9.07726,.08032,.08471,.09108,.06586,.06857,.07074,.07260,.07436,
A.07628,.07869,.08202,.08679)
DATA((C(I),I=608,714)=.07604,.08148,.08798,.1001,.07040,.07349,
1.07794,.08363,.06784,.06998,.07235,.07545,.06563,.06794,.06991,
2.07190,.06310,.06598,.06819,.07005,.06013,.06379,.06646,.06857,
3.05671,.06136,.06458,.06705,.05306,.05874,.06258,.06545,.04968,

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4.05606,.06051,.06380,.04708,.05356,.05845,.06214,.04536,.05150,
5.05653,.06053,.04429,.05002,.05491,.05904,.04360,.04911,.05369,
6.05776,.07230,.07451,.07692,.07988,.08333,.06848,.06982,.07151,
7.07354,.07604,.06620,.06740,.06862,.06990,.07133,.06373,.06543,
8.06670,.06786,.06898,.06139,.06321,.06479,.06612,.06730,.05989,
9.06047,.06256,.06423,.06563,0,.05713,.05989,.06205,.06378,0,
A.05415,.05541,.05951,.06167,0,.04699,.05278,.05658,.05931,0,
B.02675,.05111,.05333,.05671,0,.02241,.04695,.05004,.05397)
DATA((C(I),I=715,802)=0,0,.04066,.04449,.04756,.05004,.05215,
1.05397,0,0,.03848,.04174,.0446,.04711,.04931,.0513,0,0,.03638,
2.0397,.04244,.0448,.04696,.04895,0,.03002,.0339,.03800,.04085,
3.04315,.04519,.04708,.01591,.02509,.03188,.03644,.03956,.04193,
4.04392,.04571,.01583,.02367,.03023,.03504,.03844,.04098,.04301,
5.04476,.01609,.02284,.02898,.03383,.03745,.04018,.04231,.04409,
6.01665,.02249,.02814,.03288,.03661,.03949,.04176,.04360,.006768,
7.008036,.009372,.01121,0,0,.008661,.009647,.01094,.01245,.01408,
8.01585,.01057,.01130,.01224,.01337,.01462,.01596,.01246,.01303,
9.01376,.01463,.01560,.01665)
P=PRES
H=ENTH
IF(H.LT.125.0) GO TO 15
IF(H.LT.425.0) GO TO 11
IF(H.LT.4500.0) GO TO 8
IF(H.LT.9600.0) GO TO 7
IF(H.GE.20000.0)H=19999.9999
N=1
1 IF(P.LT.29.392) GO TO 4
IF(P.LT.734.80) GO TO 2
F=P/734.8-1.0
IP=5
GO TO 35
2 IF(P.LT.146.96) GO TO 3
IP=4
F=(P-146.96)/587.84
GO TO 35
3 IP=3
F=(P-29.392)/117.568
GO TO 35
4 IF(P.LT.7.348) GO TO 6
IF(P.LT.14.696) GO TO 5
IP=2
F=P/14.696-1.0
GO TO 35
5 IP=1
F=P/7.348-1.0
GO TO 35
6 IP=0
F=(P-1.4696)/5.8784
GO TO 35
7 N=2
GO TO 1
8 IF(H.GE.1025.0) GO TO 10
IF(P.LT.600.0) GO TO 9
N=4

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    GO TO 33
9  N=5
    GO TO 33
10 N=3
    GO TO 33
11 IF(H.LT.225.0) GO TO 13
    IF(P.LT.650.0) GO TO 12
    N=6
    GO TO 33
12 N=7
    GO TO 33
13 IF(P.LT.650.0) GO TO 14
    N=8
    GO TO 33
14 N=9
    GO TO 33
15 IF(P.GE.1000.0) GO TO 24
    IF(P.GE.400.0) GO TO 23
    IF(H.LT.20.0) GO TO 20
    IF(P.LT.50.0) GO TO 16
    N=17
    GO TO 17
16 N=18
17 IF(P.GE.187.506) GO TO 33
    DO 18 I=2,19
    IF(P-PL(I))19,19,18
18 CONTINUE
19 D=PL(I)-PL(I-1)
    DF=PL(I)-P
    DB=P-PL(I-1)
    HGAS=(HG(I)*DB+HG(I-1)*DF)/D
    IF(H.GE.HGAS) GO TO 33
    HLIQ=(HL(I)*DB+HL(I-1)*DF)/D
    IF(H.LE.HLIQ) GO TO 33
    CLIQ=(CL(I)*DB+CL(I-1)*DF)/D
    PHCOND=((CG(I)*DB+CG(I-1)*DF)/D-CLIQ)*(H-HLIQ)/(HGAS-HLIQ)+CLIQ
    RETURN
20 N=16
21 IF(H.GT.(-132.82+0.04*P)) GO TO 17
22 PR=P/500.0
    I=PR
    IF(I.GT.9) I=9
    F=PR-I
    HSOL=F*HS(I+2)+(1.0-F)*HS(I+1)
    IF(H.LT.HSOL) H=HSOL
    GO TO 33
23 N=15
    GO TO 22
24 IF(P.GE.3000.0) GO TO 27
    IF(H.GE.20.0) GO TO 25
    N=14
    GO TO 22
25 IF(H.GE.65.0) GO TO 26
    N=13

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      GO TO 33
26  N=11
      GO TO 33
27  IF(P.GE.4000.)GO TO 28
      IF(H.GE.65.0) GO TO 26
      N=12
      GO TO 22
28  N=10
      GO TO 22
33  FP=(P-BP(N))/DP(N)
      IP=FP
      IF(IP.GT.MX(N))IP=MX(N)
      F=FP-IP
35  FP=1.0-F
      FH=(H-BH(N))/DH(N)
      IH=FH
      FF=FH-IH
      FH=1.0-FF
      I=IH*JP(N)+IP+LOC(N)
      J=I+JP(N)
      PHCOND=FP*FH*C(I)+F*FH*C(I+1)+FP*FF*C(J)+F*FF*C(J+1)
      RETURN
      END
      SUBROUTINE PHLAMBDA
      COMMON/PHLAMB/C(802)
      DATA((C(I),I=1,122)=.3600,.3573,.3567,.3559,.3558,.3558,.3558,
1.4077,.4050,.4047,.4004,.3947,.3908,.3904,.4731,.4641,.4609,.4523,
2.4367,.4273,.4262,.5796,.5335,.5286,.5133,.4849,.4689,.4660,.7455,
3.6393,.6191,.5914,.5441,.5189,.5122,.9847,.7960,.7492,.6987,.6216,
4.5724,.561,1.2441,.9812,.9073,.8269,.7161,.6376,.619,1.5428,1.207,
51.1035,.9843,.8335,.7203,.6904,1.8668,1.4666,1.3339,1.1693,.9758,
6.8259,.7794,.2187,.2187,.2187,.2187,.2187,.2192,.2207,.3600,.3573,
7.3567,.3559,.3558,.3558,.3558,.05871,.06345,.07242,.08140,.08735,
8.09162,.09429,.09594,.09764,.09992,.1015,.1050,.1093,.1132,.1167,
9.1195,.1214,.1231,.1372,.1377,.1391,.1411,.1433,.1456,.1477,.1496,
A.1517,.1785,.1787,.1793,.1804,.1818,.1833,.1848,.1863,.1878,.2187,
B.2187,.2190,.2197,.2205,.2214,.2225,.2235,.2246)
      DATA((C(I),I=123,244)=.05660,.06723,.07289,.07710,.08113,.08490,
1.06981,.08064,.08665,.09073,.09381,.09714,.08113,.09081,.09703,
2.1012,.1038,.1062,.08789,.09606,.1020,.1063,.1089,.1110,.04041,
3.04230,.04532,.04882,.05236,.05571,.05873,.05828,.05947,.06146,
4.06394,.06661,.06938,.07198,.07286,.07366,.07504,.07683,.07883,
5.08097,.08307,.08190,.08246,.08345,.08475,.08624,.08787,.08953,
6.03899,.05601,.06901,.07673,.08122,.08482,.04023,.05794,.06989,
7.07811,.08297,.08608,.04194,.06065,.07162,.07997,.08520,.08849,
8.04415,.06382,.07408,.08225,.08779,.09130,.04683,.06723,.07710,
9.08490,.09061,.09437,.01992,.02343,.02912,.03526,.04051,.04460,
A.04769,.05005,.02481,.02738,.03166,.03675,.04158,.04570,.04905,
B.05174,.02964,.03176,.03530,.03965,.04399,.04792,.05130,.05410,
C.03466,.03643,.03939,.04316,.04708,.05078,.05408,.05693,.03964,
D.04116,.04372,.04704,.05060,.05407,.05726,.06009)
      DATA((C(I),I=245,334)=.04711,.05776,.06546,.07053,.07417,.07699,
1.07946,.08233,.08689,.09508,.04672,.05658,.06441,.06982,.07369,
2.07667,.07909,.08140,.08434,.08916,.04683,.05589,.06354,.06919,

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3.07328,.07641,.07891,.08107,.08332,.08638,.04716,.05565,.06301,
4.06887,.07311,.07624,.07882,.08099,.08297,.08521,.04750,.05601,
5.06310,.06901,.07344,.07673,.07924,.08122,.08294,.08482,.00829,
6.01665,.02814,.03661,.04176,.04518,.04788,.05030,.01156,.01801,
7.02749,.03558,.04106,.04472,.04742,.04968,.01454,.01968,.02757,
8.03505,.04061,.04451,.04732,.04954,.01731,.02151,.02817,.03496,
9.04043,.04448,.04744,.04971,.01992,.02343,.02912,.03526,.04051,
A.04460,.04769,.05005)
DATA((C(I),I=335,429)=.1447,.1674,.2,0,0,.1276,.1495,.1765,.209,0,
1.115,.1318,.1543,.1822,.2140,.1057,.1187,.1362,.1593,.1872,.09884,
2.1089,.1225,.1406,.1643,.09378,.1016,.1123,.1265,.1451,.09007,
3.09618,.1045,.1157,.1305,.08737,.09215,.09868,.1075,.1192,.08541,
4.08918,.09431,.1013,.1106,.08401,.087,.09106,.09657,.1040,.08302,
5.08542,.08865,.09303,.09891,.08233,.08429,.08689,.09038,.09508,
6.06214,.06840,.07251,.07564,.07888,.08400,.09378,.06092,.06762,
7.07198,.07520,.07807,.08186,.08861,.05976,.06687,.07148,.07482,
8.07757,.08061,.08541,.05869,.06615,.07099,.07449,.07724,.07988,
9.08347,.05776,.06546,.07053,.07417,.07699,.07946,.08233)
END

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FUNCTION PHVISC(PRES,ENTH)
COMMON/PHMU/V(768)
DIMENSION LOC(20),JP(20),DP(20),DH(20),BP(20),BH(20),MX(20),
1PL(19),HL(19),HG(19),VL(19),VG(19),HS(11)
DATA(LOC=1,17,33,180,45,75,105,123,177,222,247,297,337,357,412,437
1,497,537,633,673)
DATA(JP=4,4,3,5,5,5,3,9,5,5,5,5,5,5,5,5,8,8,8)
DATA(MX=2,2,1,0,3,3,1,7,3,3,3,3,3,3,3,3,6,6,6)
DATA(BP=0,5,1,0,0,1000,0,1000,4000,4000,3000,3000,1000,
12000,2000,1000,1000,-50,-50,-50)
DATA(DP=500,15,2,3000,750,1000,500,500,250,250,250,250,
1,500,250,250,250,250,150,150,150)
DATA(BH=8000,8000,8000,1800,425,200,200,100,15,60,-20,
130,40,-55,0,-90,-30,-130,-70,-20)
DATA(DH=4000,4000,4000,6200,275,45,45,20,5,10,5,10,20,
1,5,10,5,10,5,10,20)
DATA(PL=1.022,2,4,8,14,25,43,69,99,128,151,165,176,182
1,185,186.5,187.25,187.46875,187.506)
DATA(HL=-132.8,-129.13,-124.25,-117.79,-110.86,-101.30,-89.04,
1-74.22,-58.58,-43.43,-30.07,-20.56,-11.13,-4.27,1.17,5.54,10.83,
214.29,16.36)
DATA(HG=60.31,65.11,70.59,76.35,80.98,85.11,87.40,86.54,81.94,
174.15,64.83,56.86,47.34,39.56,33.46,28.34,22.31,18.66,16.55)
DATA(HS=-132.82,-113.35,-94.24,-75.47,-57.04,-38.91,-21.05,-3.4,
114.08,31.36,48.53)
DATA(VL=1.513E-10,1.287E-10,1.112E-10,.9093E-10,.7848E-10,.6685E-1
10,.5667E-10,.4797E-10,.4112E-10,.3581E-10,.3188E-10,.2926E-10,
2 .2688E-10,.2506E-10,.2332E-10,.2197E-10,.2132E-10,.2128E-10,
3 .2074E-10),(VG=.4274E-11,.4702E-11,.5230E-11,.5867E-11,.6488E-1
41,.7270E-11,.8189E-11,.9234E-11,.1034E-10,.1156E-10,.1292E-10,
5 .1419E-10,.1567E-10,.1700E-10,.1839E-10,.1956E-10,.2016E-10,
6 .2033E-10,.2074E-10)
DATA((V(I),I= 1,122)= 1.497E-10,1.495E-10,1.492E-10,1.490E-10,
1 1.815E-10,1.791E-10,1.765E-10,1.739E-10,2.112E-10,2.108E-10,
2 2.091E-10,2.072E-10,2.255E-10,2.314E-10,2.342E-10,2.367E-10,
3 1.497E-10,1.497E-10,1.497E-10,1.497E-10,1.804E-10,1.812E-10,
4 1.813E-10,1.813E-10,2.071E-10,2.102E-10,2.108E-10,2.112E-10,
5 2.188E-10,2.237E-10,2.252E-10,2.261E-10,1.497E-10,1.497E-10,
6 1.497E-10,1.761E-10,1.797E-10,1.804E-10,1.981E-10,2.052E-10,
7 2.071E-10,2.069E-10,2.161E-10,2.188E-10,.2298E-10,.2651E-10,
8 .3040E-10,.3422E-10,.3808E-10,.3189E-10,.3302E-10,.3473E-10,
9 .3827E-10,.4148E-10,.3798E-10,.3861E-10,.3963E-10,.4229E-10,
A .4481E-10,.4345E-10,.4381E-10,.4446E-10,.4631E-10,.4814E-10,
B .4861E-10,.4881E-10,.4921E-10,.5033E-10,.5147E-10,.5354E-10,
C .5363E-10,.5387E-10,.5435E-10,.5480E-10,.2604E-10,.3604E-10,
D .4626E-10,.5783E-10,.7155E-10,.2570E-10,.3427E-10,.4291E-10,
E .5248E-10,.6350E-10,.2586E-10,.3331E-10,.4075E-10,.4887E-10,
F .5806E-10,.2633E-10,.3288E-10,.3937E-10,.4641E-10,.5427E-10,
G .2700E-10,.3280E-10,.3854E-10,.4474E-10,.5158E-10,.2777E-10,
H .3296E-10,.3808E-10,.4360E-10,.4968E-10,.1325E-10,.1995E-10,
I .2604E-10,.1560E-10,.2067E-10,.2570E-10,.1774E-10,.2173E-10,
J .2586E-10,.1967E-10,.2292E-10,.2633E-10,.2141E-10,.2413E-10,
K .2700E-10,.2298E-10,.2532E-10,.2777E-10)
DATA((V(I),I=123,221)= .3039E-10,.3787E-10,.4532E-10,.5323E-10,

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1 .6197E-10,.7191E-10,.8349E-10,.9729E-10,1.143E-10,.2891E-10,
2 .3580E-10,.4258E-10,.4967E-10,.5737E-10,.6593E-10,.7566E-10,
3 .8689E-10,1.0005E-10,.278E-10,.3417E-10,.4039E-10,.4682E-10,
4 .5371E-10,.6123E-10,.6964E-10,.7914E-10,.8997E-10,.2699E-10,
5 .3288E-10,.3861E-10,.4449E-10,.5072E-10,.5747E-10,.6489E-10,
6 .7311E-10,.8236E-10,.2642E-10,.3188E-10,.3718E-10,.4259E-10,
7 .4828E-10,.5438E-10,.6101E-10,.6831E-10,.7637E-10,.2604E-10,
8 .3112E-10,.3604E-10,.4104E-10,.4626E-10,.5182E-10,.5783E-10,
9 .6437E-10,.7155E-10,2.187E-10,2.894E-10,0.000E-10,.5354E-10,
A .5480E-10,1.921E-10,2.457E-10,3.170E-10,1.497E-10,1.497E-10,
B 1.725E-10,2.130E-10,2.715E-10,0.000E-10,0.000E-10,1.574E-10,
C 1.888E-10,2.363E-10,3.029E-10,0.000E-10,1.455E-10,1.708E-10,
D 2.076E-10,2.644E-10,3.340E-10,1.358E-10,1.567E-10,1.857E-10,
E 2.295E-10,2.880E-10,1.277E-10,1.455E-10,1.690E-10,2.026E-10,
F 2.512E-10,1.208E-10,1.362E-10,1.559E-10,1.826E-10,2.219E-10,
G 1.149E-10,1.284E-10,1.453E-10,1.672E-10,1.978E-10)
DATA((V(I),I=222,336)= 1.097E-10,1.218E-10,1.365E-10,1.550E-10,
1 1.796E-10,1.011E-10,1.110E-10,1.226E-10,1.366E-10,1.540E-10,
2 .9413E-10,1.026E-10,1.122E-10,1.234E-10,1.367E-10,.8837E-10,
3 .9576E-10,1.040E-10,1.133E-10,1.240E-10,.8349E-10,.9007E-10,
4 .9729E-10,1.053E-10,1.143E-10,2.061E-10,2.623E-10,0,0,0,1.787E-
5 10,2.353E-10,3.116E-10,0,0,1.590E-10,2.025E-10,2.565E-10,0,0,
6 1.441E-10,1.773E-10,2.297E-10,2.901E-10,0.000E-10,1.325E-10,
7 1.589E-10,1.989E-10,2.523E-10,0.000E-10,1.230E-10,1.448E-10,
8 1.758E-10,2.241E-10,2.742E-10,1.152E-10,1.336E-10,1.585E-10,
9 1.954E-10,2.509E-10,1.086E-10,1.245E-10,1.452E-10,1.742E-10,
A 2.187E-10,1.030E-10,1.169E-10,1.345E-10,1.581E-10,1.921E-10,
B .9807E-10,1.104E-10,1.257E-10,1.454E-10,1.725E-10,.9376E-10,
C 1.049E-10,1.183E-10,1.352E-10,1.574E-10,.8649E-10,.9578E-10,
D 1.066E-10,1.196E-10,1.358E-10,.8058E-10,.8857E-10,.9767E-10,
E 1.082E-10,1.208E-10,.7565E-10,.8268E-10,.9053E-10,.9944E-10,
F 1.097E-10,.7147E-10,.7775E-10,.8467E-10,.9239E-10,1.011E-10,
G .6786E-10,.7355E-10,.7975E-10,.8657E-10,.9413E-10,.6472E-10,
H .6992E-10,.7555E-10,.8166E-10,.8837E-10,.6197E-10,.6676E-10,
I .7191E-10,.7746E-10,.8349E-10)

```

P=PRES

H=ENTH

IF(H.LT.100.0) GO TO 9

IF(H.LT.425.0) GO TO 5

IF(H.LT.8000.0) GO TO 3

IF(H.GE.20000.) H=19999.9999

IF(P.LT.50.0) GO TO 1

N=1

GO TO 33

1 IF(P.LT.5.0) GO TO 2

N=2

GO TO 33

2 N=3

GO TO 33

3 IF(H.LT.1800.0) GO TO 4

N=4

GO TO 33

4 N=5

GO TO 33

```

5 IF(H.LT.200.0) GO TO 7
  IF(P.LT.1000.0) GO TO 6
  N=6
  GO TO 33
6 N=7
  GO TO 33
7 IF(P.LT.1000.0) GO TO 8
  N=8
  GO TO 33
8 N=20
  GO TO 33
9 IF(P.LT.3000.0) GO TO 13
  IF(P.LT.4000.0) GO TO 11
  IF(H.GE.60.) GO TO 10
  N=9
  GO TO 30
10 N=10
  GO TO 33
11 IF(H.GE.30.) GO TO 12
  N=11
  GO TO 30
12 N=12
  GO TO 33
13 IF(P.LT.1000.0) GO TO 18
  IF(H.LT.40.) GO TO 14
  N=13
  GO TO 33
14 IF(P.LT.2000.0) GO TO 16
  IF(H.GE.0.) GO TO 15
  N=14
  GO TO 30
15 N=15
  GO TO 33
16 IF(H.GE.-30.0) GO TO 17
  N=16
  GO TO 30
17 N=17
  GO TO 33
18 IF(H.GE.-70.0) GO TO 19
  N=18
  IF(H.GT.(-132.82+0.04*P)) GO TO 22
  GO TO 30
19 IF(H.GE.-20.0) GO TO 20
  N=19
  GO TO 22
20 N=20
22 IF(P.GE.187.506) GO TO 33
  DO 23 I=2,19
  IF(P-PL(I))24,24,23
23 CONTINUE
24 D=PL(I)-PL(I-1)
  DF=PL(I)-P
  DB=P-PL(I-1)
  HGAS=(HG(I)*DB+HG(I-1)*DF)/D

```

```

IF(H.GE.HGAS) GO TO 33
HLIQ=(HL(I)*DB+HL(I-1)*DF)/D
IF(H.LE.HLIQ) GO TO 33
VLIQ=(VL(I)*DB+VL(I-1)*DF)/D
PHVISC=((VG(I)*DB+VG(I-1)*DF)/D-VLIQ)*(H-HLIQ)/(HGAS-HLIQ)+VLIQ
RETURN
30 PR=P/500.0
I=PR
IF(I.GT.9) I=9
F=PR-I
HSOL=F*HS(I+2)+(1.0-F)*HS(I+1)
IF(H.LT.HSOL) H=HSOL
33 FP=(P-BP(N))/DP(N)
IP=FP
IF(IP.GT.MX(N)) IP=MX(N)
F=FP-IP
FP=1.0-F
FH=(H-BH(N))/DH(N)
IH=FH
FF=FH-IH
FH=1.0-FF
I=IH*JP(N)+IP+LOC(N)
J=I+JP(N)
PHVISC=FP*FH*V(I)+F*FH*V(I+1)+FP*FF*V(J)+F*FF*V(J+1)
RETURN
END
SUBROUTINE PHDTMU
COMMON/PHMU/V(768)
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1 .8649E-10,.3483E-10,.4377E-10,.5308E-10,.6347E-10,.7565E-10,
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3 .3787E-10,.4532E-10,.5323E-10,.6197E-10,1.827E-10,2.403E-10,
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510,0,0,1.257E-10,1.581E-10,2.122E-10,2.758E-10,0,1.151E-10,1.409E-
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7 2.098E-10,2.609E-10,.9958E-10,1.175E-10,1.422E-10,1.800E-10,
8 2.409E-10,.9366E-10,1.091E-10,1.296E-10,1.590E-10,2.061E-10,
9 .8859E-10,1.021E-10,1.195E-10,1.433E-10,1.787E-10,.8419E-10,
A .9627E-10,1.113E-10,1.311E-10,1.590E-10,.8031E-10,.9122E-10,
B 1.045E-10,1.214E-10,1.441E-10,.7688E-10,.8681E-10,.9868E-10,
C 1.134E-10,1.325E-10,.7103E-10,.7949E-10,.8928E-10,1.009E-10,
D 1.152E-10,.6621E-10,.7360E-10,.8196E-10,.9159E-10,1.030E-10,
E .6216E-10,.6874E-10,.7605E-10,.8429E-10,.9376E-10,.5870E-10,
F .6465E-10,.7115E-10,.7837E-10,.8649E-10)
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210,1.320E-10,1.830E-10,2.446E-10,0.000E-10,.9401E-10,1.174E-10,
3 1.544E-10,2.127E-10,3.000E-10,.8694E-10,1.063E-10,1.348E-10,
4 1.831E-10,2.438E-10,.8108E-10,.9759E-10,1.205E-10,1.560E-10,
5 2.131E-10,.7611E-10,.9049E-10,1.096E-10,1.372E-10,1.827E-10,
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9 .9679E-10,1.151E-10,.5919E-10,.6809E-10,.7840E-10,.9085E-10,

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A 1.066E-10,.5461E-10,.6242E-10,.7120E-10,.8138E-10,.9366E-10,
B .5075E-10,.5778E-10,.6546E-10,.7412E-10,.8419E-10,.4746E-10,
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3, 1.158E-10,1.464E-10,1.863E-10,2.795E-10,0,0,.715E-10,.8467E-10,
4 1.007E-10,1.225E-10,1.550E-10,1.943E-10,2.307E-10,0.000E-10,
5 .6556E-10,.7668E-10,.8965E-10,1.063E-10,1.293E-10,1.637E-10,
6 2.014E-10,0.000E-10,.6057E-10,.7025E-10,.8117E-10,.9458E-10,
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H .3000E-10,.3781E-10,.4308E-10,.4840E-10,.5374E-10,.5931E-10,
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K .4094E-10,.4509E-10,.4965E-10,.5431E-10,.5919E-10,0,.2690E-10,
L .3246E-10,.3731E-10,.4169E-10,.4594E-10,.5021E-10,.5461E-10)
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2 .2296E-10,.2787E-10,.3185E-10,.3543E-10,.3882E-10,.4213E-10,
3 0.000E-10,.1356E-10,.1992E-10,.2471E-10,.2852E-10,.3188E-10,
4 .3502E-10,.3803E-10,.0073E-10,.1111E-10,.1768E-10,.2239E-10,
5 .2599E-10,.2914E-10,.3205E-10,.3483E-10,.0331E-10,.1040E-10,
6 .1614E-10,.2070E-10,.2412E-10,.2706E-10,.2976E-10,.3233E-10,
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8 .2802E-10,.3039E-10,.0702E-10,.1131E-10,.1506E-10,.1869E-10,
9 .2179E-10,.2438E-10,.2671E-10,.2891E-10,.0857E-10,.1207E-10,
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C .2510E-10,.2699E-10,.1129E-10,.1380E-10,.1607E-10,.1832E-10,
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E .1668E-10,.1862E-10,.2062E-10,.2257E-10,.2438E-10,.2604E-10)
END

```

1.2 P-V-T Measurements of Oxygen

Installation of the apparatus in its new quarters has been completed. The nuisance volumes have been redetermined, inasmuch as these have incurred small changes. Runs have been made that overlap some of the earlier series in order to establish repeatability.

2. Cryogenic Metrology (Instrumentation)

2.0 General Comments

Personnel contributing to the activities during this period were: S. B. Lang, J. W. Dean, T. M. Flynn, and R. J. Richards.

2.1 Temperature

2.1.1 Experimental Pyroelectric Studies

Experimental data for the calculation of the pyroelectric coefficient, dc dielectric constant, and volume resistivity of several ferroelectric ceramic materials were taken. Preliminary results for the pyroelectric coefficient of Clevite Ceramic B (95% BaTiO_3 , 5% CaTiO_3) are shown in Figure 1. The rhombohedral-orthorhombic phase transition at 155-170°K, the orthorhombic-tetragonal phase transition at 240-255°K, and the Curie point tetragonal-cubic phase transition) at 387°K are especially prominent. The characteristic hystereses in the transition temperatures as a function of direction of temperature change can also be observed. The phase transitions produced noticeable peaks in the dielectric constant values also. Additional data in the region 4 to 78°K have been measured for Clevite Ceramic B, but are not shown in Figure 1.

Measurements have also been taken over the temperature range 4 to 300°K of the properties of Clevite PZT-4 and PZT-5 (polarized lead-zirconate-titanate ceramics). No phase-transitions were observed in either material over this range. In addition, the pyroelectric coefficients of both materials were considerably higher than those of Ceramic B. For these two reasons, PZT-4 and PZT-5 are probably more suitable as pyroelectric thermometer sensors than Ceramic B. Quantitative results will be presented in the next report.

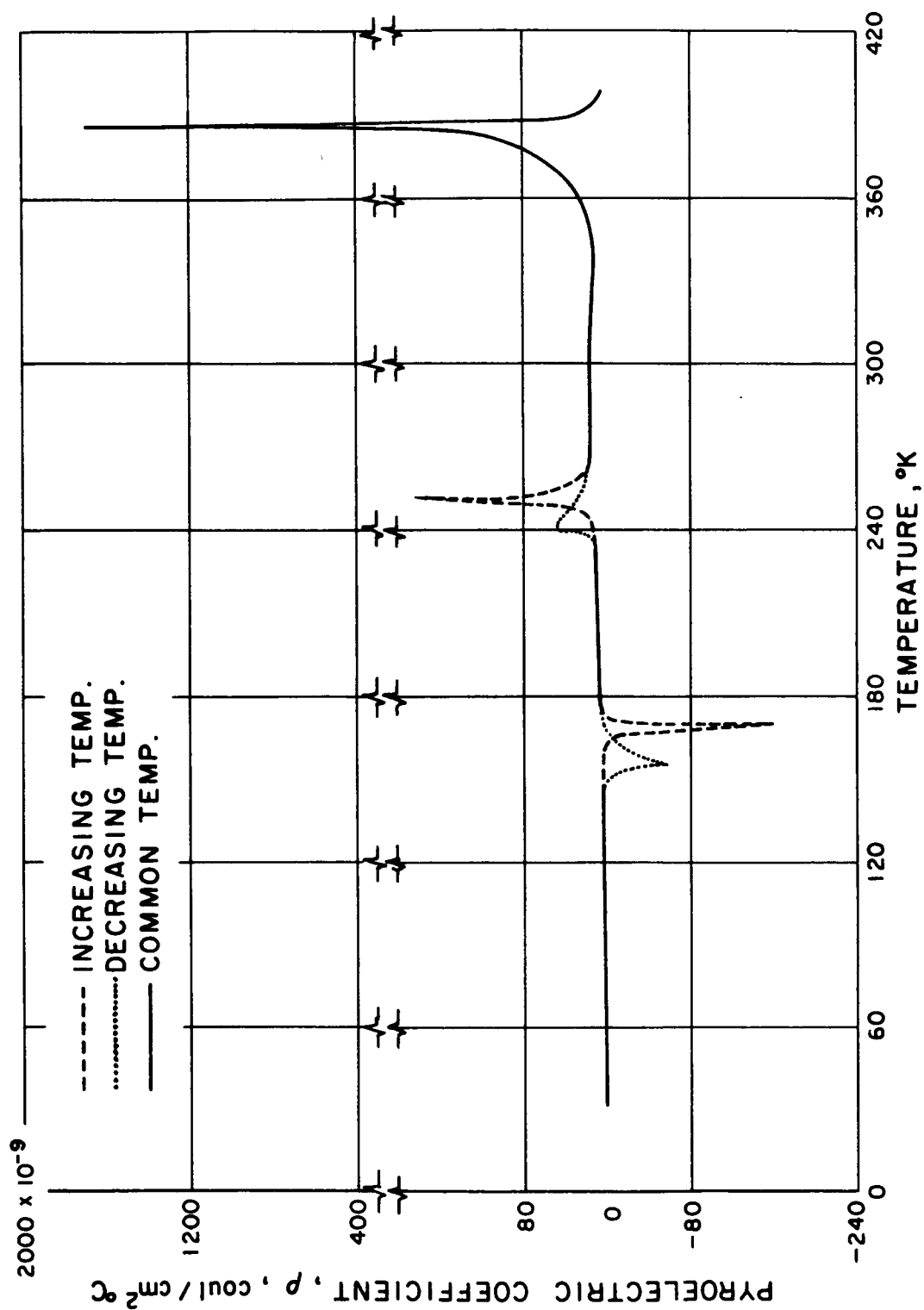


Fig. 1 Pyroelectric Coefficient of Clevite Ceramic B
 Electrodes are normal to tetragonal polar axis

2.1.2 Publications

A description of the equipment, method of calculation, and preliminary results on Ceramic B have been published by Steven A. Shaw this year as a Master of Science thesis presented to the Dept. of Chemical Engineering of the University of Colorado. The thesis title is "Measurement of the Characteristics of Pyroelectric Crystals for Thermometric Purposes with Special Reference to Barium Titanate Ceramic".

2.2 Pressure

2.2.1 Piezoresistive Effects in Materials

An apparatus has been constructed to study the piezoresistive effects in materials over a wide range of temperatures. A sample chamber is located at the end of a vacuum insulated probe in such a manner that the probe may be inserted into a dewar containing either liquid nitrogen, hydrogen, or helium. The vapor pressure of the cryogen is maintained with a Cartesian diver type of regulator. Since the sample is housed in a copper holder, its temperature is held constant. Gas pressure up to 5000 psi may be admitted into the sample holder while the sample's resistance is monitored by a potentiometric method.

Preliminary investigations have been made with carbon. Figure 2 shows the resistance dependency of carbon as a function of pressure at several temperatures. The room temperature and nitrogen temperature results are as expected; however, the hydrogen temperature results show a reversal in slope. More tests will be done to verify this effect; however, if true, this diminishes the usefulness of carbon as a pressure sensor over a wide temperature range.

Samples of rare earth metals have been ordered for further investigation.

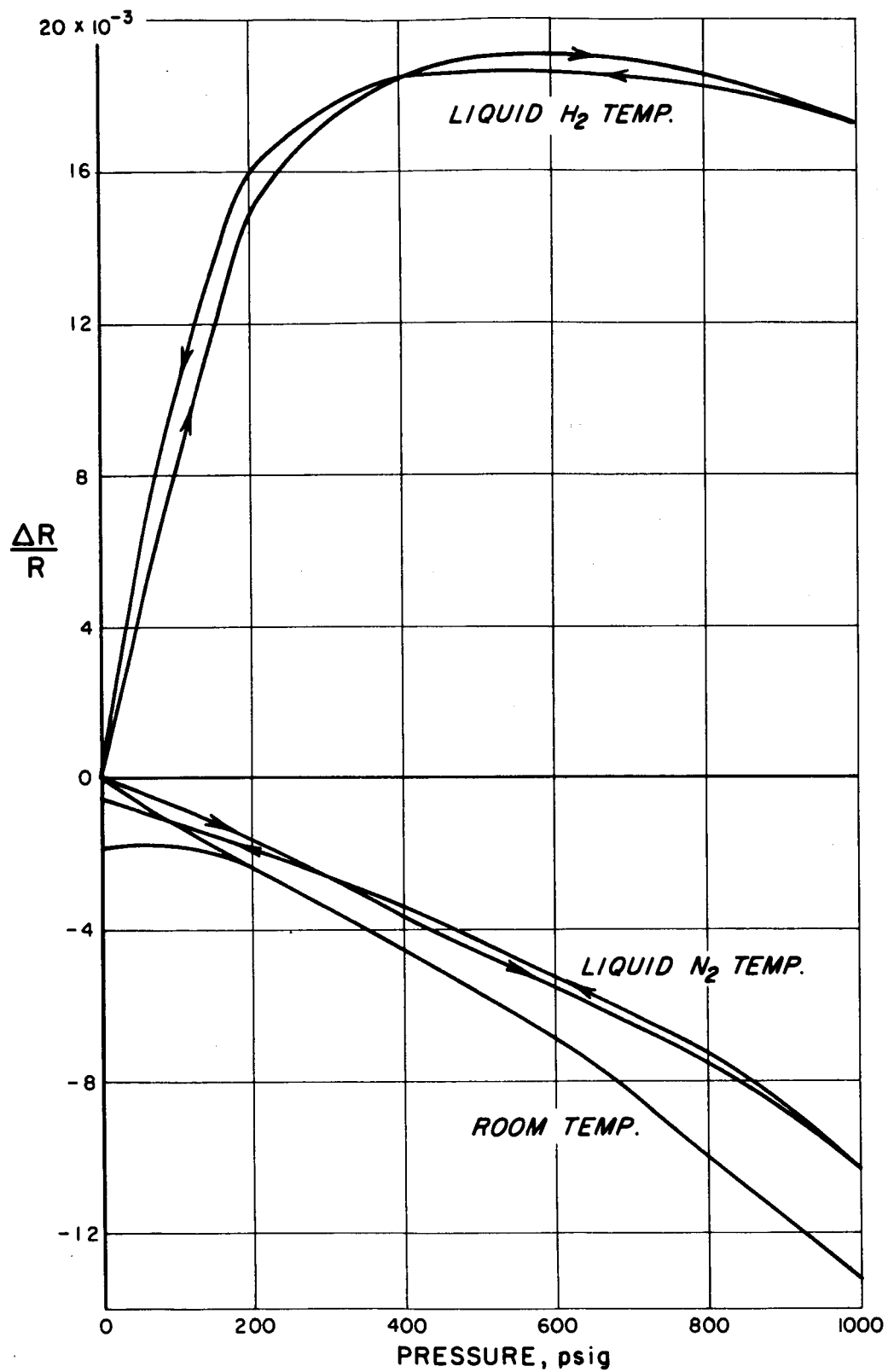


Fig. 2 Resistance Change of a Carbon Resistor (1000 ohm, 1/4 watt) vs Pressure at Constant Temperatures

3. Consultation and Advisory Services

Consultation and advisory services in the general field of cryogenic engineering has continued in several NASA program areas: Centaur (funded separately), Rover, and NERVA.

3.1 Centaur Program - Robert W. Arnett

Contact with NASA Lewis Research Center personnel has been frequent through the medium of telephone calls and one trip to Cleveland.

3.1.1 Stratification and Pressurization

Following the rearrangement of the complete program into a set of subroutines, debugging has progressed more rapidly. Some adjustments in the program were required following debugging. Initial runs of the complete program are in progress at the end of the reporting period.

3.1.2 Flight Data Analysis

Preparation for analysis of flight data from forthcoming launches has been completed and checked out using sample tapes furnished by NASA-LeRC. A decision was made to utilize the first digitized tapes produced from the analog tapes since extracting data on only a few sensors should be more readily accomplished. At the end of the reporting period all preparations had been made for processing of the AC-8 launch data.

3.1.3 General Dynamics/Convair Testing Program

Testing and evaluation of the liquid-vapor detectors and the temperature transducers were completed as planned. An informal report covering the response time, sensitivity, and calibration results was prepared. This report was delivered to Cleveland and an oral presentation of the testing program was presented.

Plans were made for further testing of the sensors to evaluate response to cyclic immersion, heater voltage, and

orientation. Due to manpower limitations and the greater urgency of the flight data analysis and the stratification program, this additional testing has been delayed.

3.2 Rover Program - Alan F. Schmidt

A Test Cell "C" Facility Review Meeting at the Nuclear Rocket Development Station, Nevada, was attended on January 24-26, 1966.

The purpose of this meeting was to review design, checkout, and operating procedures for the modified NRDS facility. Written comments on this review were furnished LASL upon invitation by the host organization.

Information was supplied to LASL at later dates on the thermal expansivities of beryllium and stainless steel, electrical resistivities of several fluids, and a set of documents relating to the martensite transformation of austenitic stainless steels.

3.3 NERVA Program - Alan F. Schmidt

At the request of SNPO-C, meetings were attended at the Aerojet-General Corporation, Sacramento, California on January 5-6 with the intent of determining feasibility and safety requirements for a 250 lb./sec. hydrogen burn pond to be used in conjunction with Phoebus-2 test stand H-4B. A 1/26 scale model pond has been used to provide information useful to the full scale facility, and it was hoped that data from the small pond could be extrapolated to any practical design problem or condition involving safe, economical hydrogen gas disposal, since conventional commercial flare stacks are expensive to procure, expensive to operate, and at times hazardous when manifolded or used at off-design conditions. Additional research will be required to provide the general design information required, and it appeared uncertain at the Sacramento meeting whether time or funding could be made available to complete the job initially contemplated.

On February 24, the General Dynamics/Fort Worth Reactor Facility was visited for discussions relating to cryogenic thermal conductivity experiments in a nuclear radiation field, and a para-to-ortho hydrogen conversion investigation in the same environment. Beryllium thermal conductivity test specimens previously measured at NBS-CD have been supplied GD/FW for inclusion as check pieces in each thermal conductivity cryostat. Information has been provided GD/FW and AGC/REON on appropriate thermocouple materials for these tests, thermocouple calibration problems, tables, etc. Throughout the quarter, additional communication has occurred with GD, AGC, and SNPO-C by letter and phone.

A NERVA Instrumentation Review Meeting was attended at AGC/REON on March 28-29; a trip report covering NBS impressions and commentary on this business was prepared subsequently and forwarded to SNPO-C as a matter of mutual interest.

At the request of SNPO-C, a draft copy of the Bureau of Mines hydrogen safety manual was reviewed and discussed. Also, evaluation was made of a specific hydrogen safety problem concerning Engine Stage Test Stand 2/3.

At the request of NASA-LeRC (through SNPO), information was gathered and sent to the Northwestern Technological Institute (under NASA contract) concerning the available experimental data on the thermal conductivity of fluid hydrogen.

4. Cryogenic Flow Processes

4.0 General Comments

Personnel contributing to the project during the present reporting period were: W. G. Steward, E. G. Brentari, R. V. Smith, J. A. Brennan, D. K. Edmonds, Jr., and R. Zarate.

4.1 Experimental

The two-phase choking flow experiments were completed during this period on both hydrogen and nitrogen. A preliminary report was written and submitted for possible inclusion in the Cryogenic Engineering Conference to be held in Boulder, Colorado, on June 13-14, 1966. The abstract of the report reads as follows:

Data on critical mass flow are given in this paper from experiments using a straight pipe with a vacuum jacket. On some of the runs, pressure recoveries which were interpreted as shocks were occurring slightly upstream from the exit plane. There is some indication that exit plane parameters are not directly controlling mass flow; it still seemed advisable, however, to follow current practice and report exit plane pressures and qualities. Results from simple analytical models to predict mass-limiting flow are compared with experimental results at the exit plane.

Representative graphs of the data are included for both hydrogen and nitrogen (figures 3 and 4).

Work on transfer line chilldown was resumed during this reporting period with the globe valve configuration and a partially precooled transfer line. These tests were done in order to get more comparable data between the two types of valves that had been used in previous tests (see Figure 2, page 38 of the Eighteenth Progress Report). Results of all the tests with the globe valve are shown in figure 5. Since

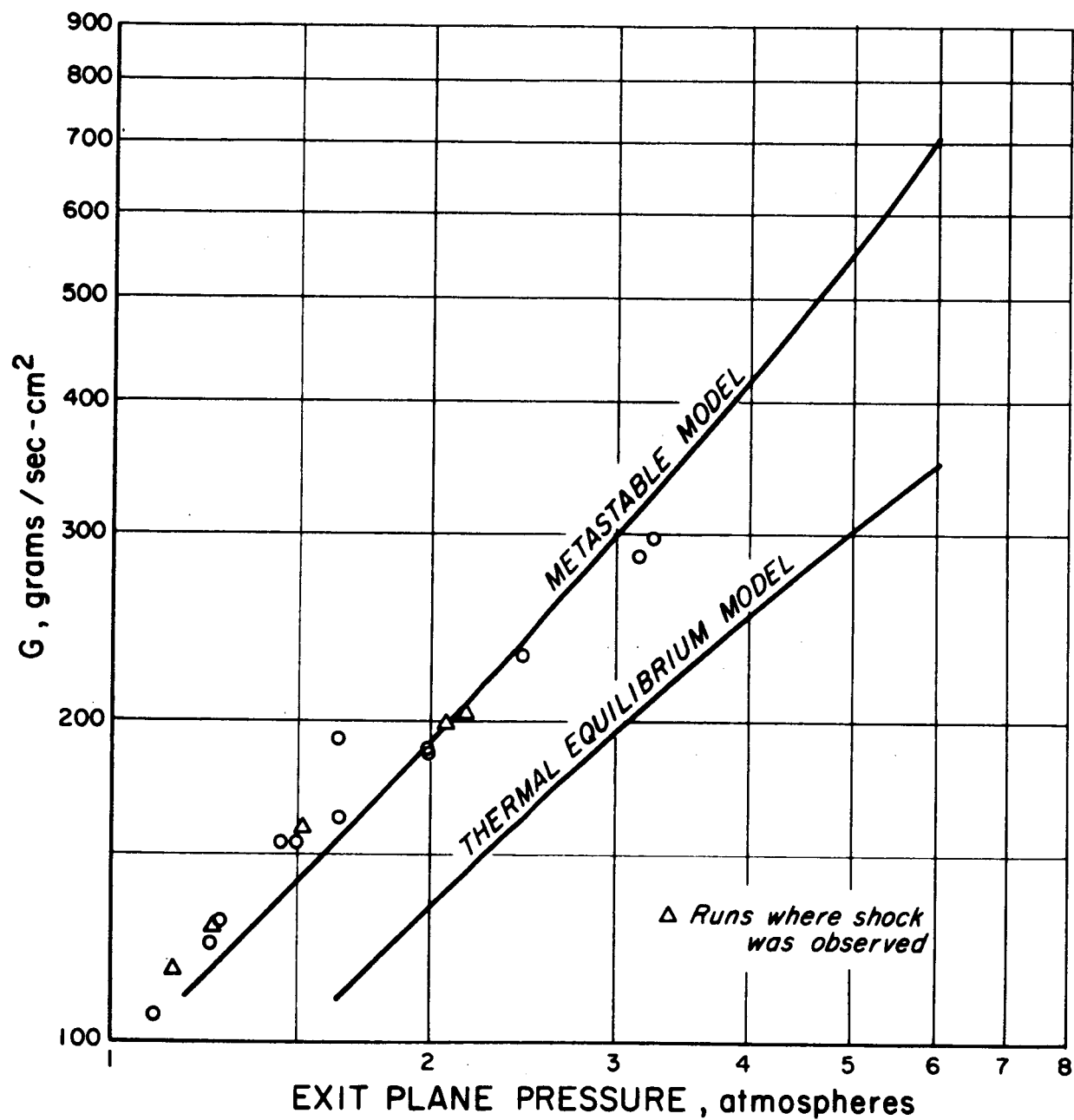


Fig. 3. Comparison of predicted values with experimental data for critical flow of liquid and gaseous hydrogen mixtures. Mass qualities from 25 through 30%.

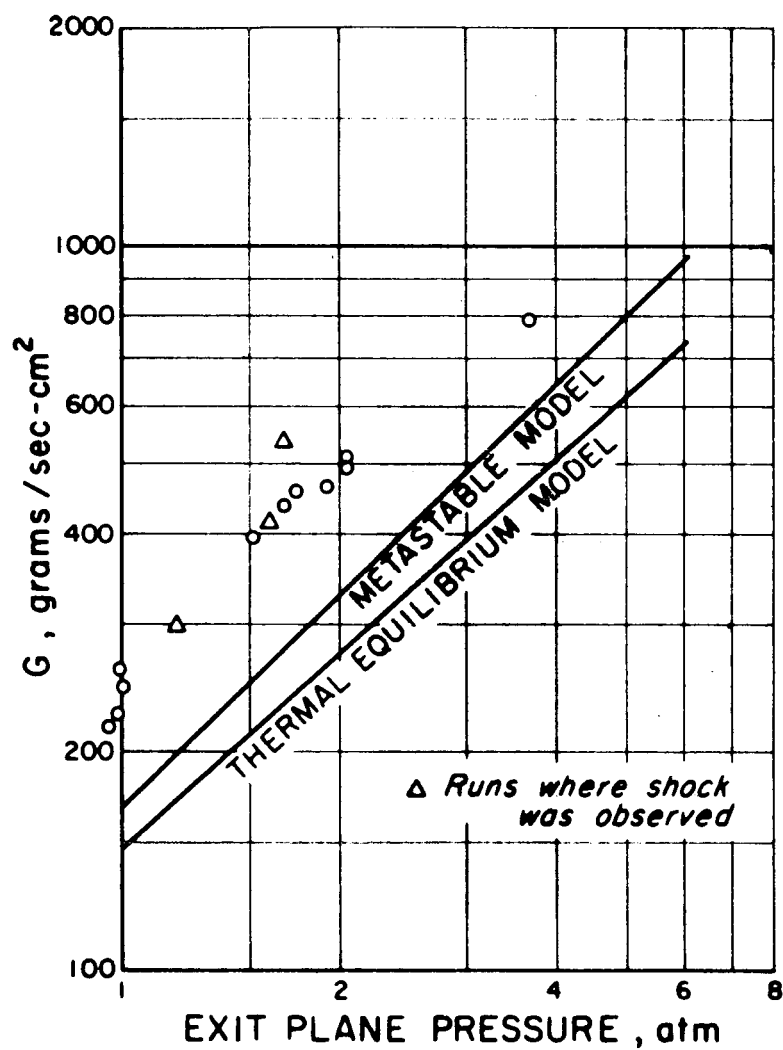


Fig. 4. Comparison of predicted values with experimental data for critical flow of liquid and gaseous nitrogen mixtures. Mass qualities from 15 through 20%.

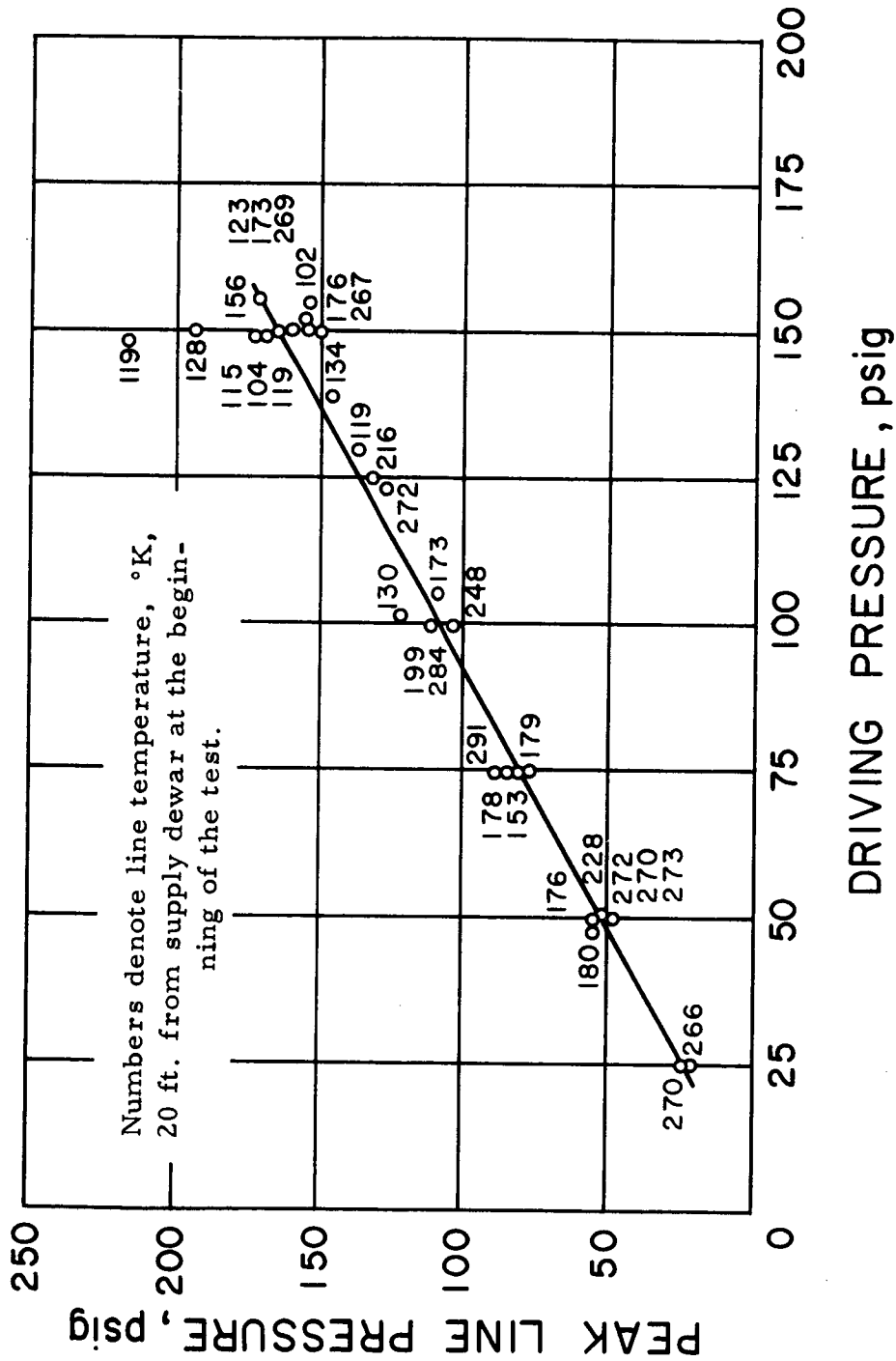


Fig. 5. Peak line pressure as a function of driving pressure for subcooled liquid hydrogen (hydrogen temp. approximately 19 1/2°K).

there was some surging experienced in the most recent tests, more experimental work is planned in an effort to establish conditions causing surges. It is apparent from the results that there is more involved than simply the line temperature 20 ft. from the supply dewar.

The data from the chilldown tests completed thus far are being put in final form for publication and should be finished during the next quarter.

4.2 Analytical

The computational scheme outlined in the last progress report has been programmed in Fortran in a basic form and is in the process of being debugged. It has not run as yet.

The basic form is for a transfer line of uniform cross-sectional area, a uniform wall, and constant driving pressure. It includes temperature dependent properties and the use of unsteady-state conservation equations for homogeneous single-phase and two-phase fluids.

An attempt has been made to keep the program as flexible as possible by dividing it into a number of subroutines. The program may then in some cases be adapted to other systems by altering one or a few subroutines.

5. Solid-Liquid Hydrogen Studies

5.0 General Comments

Personnel Contributing to the project during the present reporting period were: D. B. Mann, D. E. Daney and D. B. Chelton.

5.1 Mass Energy Balance Experiment

This portion of the program, designated as Task I (see NBS Report 9145), has been completed. A description of the experimental apparatus, experimental procedures, and results are available upon request as NBS Report 9189. The abstract and conclusions which summarize the findings follow:

Abstract (NBS Report 9189)

A theoretical and experimental investigation of the feasibility of predicting the quality of liquid-solid hydrogen mixtures from the mass fraction of vapor pumped off in the freeze-thaw process has been completed. Three independent methods of experimental quality determinations were used to check the correctness of the qualities predicted from the measured mass fraction pumped off in forming liquid-solid mixtures. In all cases only freshly made mixtures were used.

It is suggested that an independent means of determining the edge of the triple point region, such as measurement of the vapor pressure, be used. With this modification, measurement of the mass fraction pumped off during the freeze-thaw process provides a simple, non-destructive and accurate method of bulk quality determinations. The method is not appropriate for cases in which a partial transfer from the dewar is made and it requires accurate knowledge of the heat leak if long storage times are to be used. Finally, since the method is only as accurate as the accuracy of the flow and liquid volume measurements, possible low accuracy in large volume gas flowmeters places a restriction on the method.

Conclusions (NBS Report 9189)

Prediction of the quality by measuring the mass fraction of the gas pumped off appears to be an accurate and practical method of quality measurement. The experimentally measured qualities agree with the predicted qualities to within 2 percent. No significant irreversibilities appear to occur in the freeze-thaw process. The primary sources of error in this method are the errors in the flow measurement and the liquid volume measurement.

An example may aid in giving an idea of the accuracy which can be obtained by this method. For a flowmeter accurate to ± 2 percent and a liquid volume measurement accurate to ± 2 percent, the maximum uncertainty in the quality of a 0.50 solid mixture would be 0.030, 0.010 of this being the maximum uncertainty if the flow and volume are known exactly. This 0.030 uncertainty in the quality contributes an uncertainty of 0.36 percent in the determination of the total mass held in a container. For a container holding 10,000 gallons, a maximum error in the estimated weight of 24 pounds would result.

In conclusion, measurement of the mass fraction pumped off during the freeze-thaw process provides a simple, non-destructive and accurate method of quality determination. It offers the following advantages:

1. It requires the measurement of only
 - a) the mass of the vapor pumped off,
 - b) the liquid mass either before or after pumping, and
 - c) the heat leak into the dewar.
2. It is non-destructive, i. e., it does not require melting the slush.
3. It requires no viewports or apparatus inside the dewar, with the exception of a liquid level indicator.
4. It does not require a homogeneous distribution of the solid.

Since this method measures the bulk quality, it is not good for cases in which a partial transfer from the dewar is made. If the slush is stored for long periods, the uncertainty in the quality will be increased due to uncertainties in the heat leak into the dewar. Finally, since the method is only as accurate as the accuracy of the flow and liquid volume measurement, possible low accuracy in large volume gas flowmeters places a restriction on the method.

5.2 Project Design

Initial detailed planning has been started on Tasks two and three.

6. Thermal Conductivity of Solids

6.0 General Comments

Personnel contributing to the project during the present reporting period were: D. H. Weitzel, R. L. Powell, R. H. Kropschot, and John Morgan.

6.1 General Program

Activation of a Thermal Conductivity of Solids program has begun. The basic method used by R. L. Powell, et al., in previous work at this laboratory will be maintained. This comprises axial heat flow along a solid cylindrical sample, with 8 temperature measuring stations spaced at 1-inch intervals along the sample length.

Chromel vs. gold with 0.07 atomic percent iron will be used for all thermocouples. Accurate calibration data for these new thermocouples will be obtained in a separate program conducted by L. Sparks. Thermocouple potentials will be read with a precision seven-dial potentiometer and nanovolt electronic null detector. The thermocouple reference junction will be at the heat sink and monitored by a platinum resistance thermometer. At the lower temperatures the warm end of the sample will be monitored by a germanium resistance thermometer as a check on the thermocouple readings. The heat sink will be cooled successively by liquid nitrogen, liquid hydrogen, and liquid helium. Final tempering of all thermocouple leads will be carried out on a semicircular shield which parallels the sample and is automatically driven to the same temperature profile as the sample.

Several methods of radiation shielding are under consideration; through use of effective radiation shielding we hope to extend the upper temperature limit of the measurements to near room temperature. We may also isolate and control the temperature of the heat sink block at the top of the sample. This would utilize a temperature control unit

similar to that planned for the tempering shield. The mechanical modification required to accomplish this would be minor and reversible, i. e., the experiment could be run with or without the floating heat sink. Some improvement in control of heat sink temperature will in any case be achieved because we have incorporated a variable precision manostat to control vapor pressure of the cryogen. This will make the sink temperature independent of atmospheric pressure variations, and will also allow us to operate the sink at temperatures somewhat lower than the normal boiling temperatures of the cryogens.

The first material to be measured will be Titanium-5Al-2.5Sn, a well characterized sample of which has been provided by Titanium Metals Corporation of America. We plan to measure one to three other alloys of special interest to NERVA before we start work on the standard reference materials. According to our present information the next choices of alloys should be Inconel 718, Steel A-286, Inconel X, and possibly an as yet unspecified aluminum alloy to be used for the NERVA II pressure vessel.

For the standard reference materials we are considering the following: (1) High Conductivity - Platinum or Tungsten, (2) Intermediate - Copper alloy or Aluminum alloy, (3) Low Conductivity - Iron, Nickel, or Inconel.

All measurement and control instruments are either on hand or on order. Mechanical construction is about 70% complete. Wiring and assembly of panels will begin in about two weeks. This final phase will take at least a month, giving July 1 as an optimistic estimate for beginning the measurements. R. L. Powell will in the interim work out computer programs for processing the experimental data.